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Cross Sectional TEM Characterization of Epitaxial Silicon Film Grown using Hot Wire Chemical Vapor Deposition

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Abstract: We have investigated epitaxial growth of poly crystalline intrinsic silicon film grown on glass and Si/SiO_2 substrate using hot wire chemical vapour deposition technique. We have grown 20 nm nucleation layer at 400°C followed by an epitaxial growth of 200 nm thick at 600°C. Than 800 nm thick layer was grown on top using high hydrogen silane ratio of 15:5. Hydrogen soaking was performed for well passivated film. Evaluation of different layers was performed using cross sectional transmission electron microscopy. Poly crystalline as well as epitaxial and columnar growth regions were well observed.

Keywords: Cross section TEM, HWCVD, Poly crystalline silicon, Epitaxial growth.

1 Introduction

Hot-wire chemical vapour deposition (HWCVD) has been shown to be a promising technique for growing hydrogenated amorphous silicon films at low (~ 250°C) substrate temperature [1]. Low-temperature (400°C to 600°C) epitaxial thick films were reported using HWCVD [2,3,4]. We are developing oriented crystalline silicon technology on inexpensive foreign substrates for photovoltaic. Silicon is abundant, non-toxic and highly producible, but the wafer presently accounts for about half the photovoltaic module cost. Our goal to grow an alternative silicon film with the efficiency of crystal silicon and a low cost structure like amorphous silicon. Epitaxial silicon films grown on high-quality seed layers can have good crystalline quality. Thicknesses between 2 and 10 microns with excellent light trapping will be required in this case. This technology requires high-rate, high-quality silicon in 600°C temperature to which low-cost substrates such as borosilicate glass can be heated for hours without significant deformation. We have demonstrated hot-wire chemical vapor deposition (HWCVD) epitaxial growth from silane onto Si/SiO_2 and glass. With HWCVD epitaxy at about 600°C, we have grown layers up to one micron thick at 7 A° / sec on these substrates.

2 Experimental

We have used 1cm × 2cm alkali free borosilicate glass and 500 nm oxide layer on n-type 200 micron (100) silicon wafer (Si/SiO_2) as substrate. Thin 20 nm nucleation layers were grown on glass substrate at 400°C with filament temperature 1900°C along a gas ratio of SiH_4:H_2 = 1:20 for 100 sec. For thickening stage a mixture of SiH_4 and H_2 were used as process gas with a ratio of SiH_4:H_2=5:15 for 20 min. The epitaxial growth was performed in a ramp run where silane concentration increased gradually from 1sccm to 5sccm in four stages. In some stages H_2 gas flow was reduced to keep process pressure constant. We have annealed the film at this stages under 20 sccm of H_2 flow for 30min followed by a H_2 soaking during cooling the sample from growth temperature to a lower temperature of 200°C for 45 min. This stage of hydrogen passivation is introduced to improve the material quality. A thin 100 nm layer of amorphous intrinsic poly silicon films were grown at this 200°C for 25 sec using a gas ratio of SiH_4:H_2=2.5:20.

3 Characterization

A. Cross sectional transmission electron microscopy (X-TEM)

The structure of the devices were evaluated by cross sectional transmission electron microscopy (X-TEM) equipped with a field emission electron gun operated at 200 kV. We have used intrinsic silicon film grown on Si/SiO_2

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substrate to prepare X-TEM imaging sample. The sample prepared for X-TEM imaging was performed using a route explained in brief. We have deposited of 20 nm Chromium followed by 80 nm gold using thermal evaporator and cut 4×4 mm two pieces of sample using ultrasonic cutter. Than we have staked two pieces of samples facing towards each other with two more pieces of silicon wafer of same size on both sides of this structure. We have cut 700 micron thick disk from 2.3 mm diameter and 5mm long brass cylinder using disk cutter. Using manual grinder and lapping paper (45 microns for lapping and 5 micron for polishing) reduced the thickness of the disk to 100 micron. Than we have placed this 100 micron thick disk in a dimpling instrument to make a dimple around 70 microns in the middle of the disk. This part of the sample became 30 microns thick. The final step of the sample preparation is performed in a precision ion polishing system where the sample is being bombarded by Argon ion with energy of 5 kV. The duration of exposer required for the thinning depends on the sample material. One can thin the sample as thin as 100 nm which allows electron to pass through the sample for X-TEM imaging [5,6,7].

4 Results and Discussion

A. Cross section transmission electron microscopy (X-TEM)

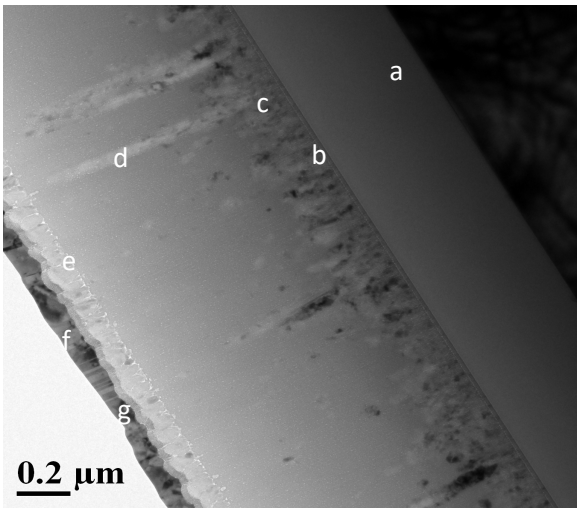


Fig. 1: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) SiO_2 layer, b) nucleation layer, c) epitaxial region, d) columnar growth, e) amorphous silicon, f) chrome, g) gold layer.

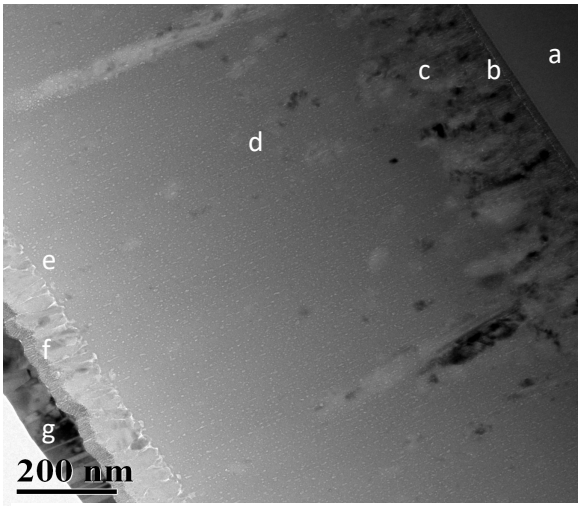


Fig. 2: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer.

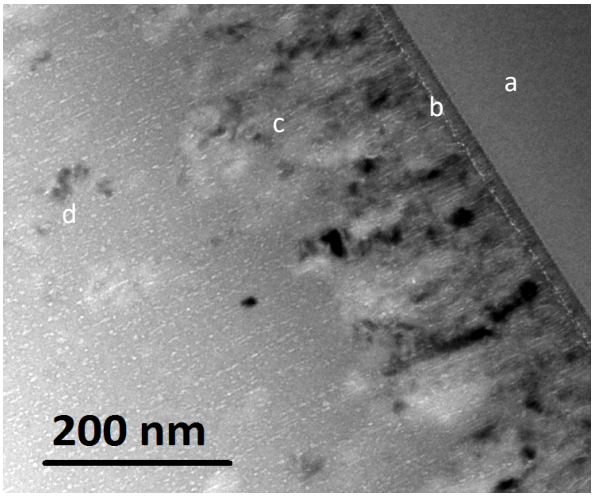


Fig.3: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) SiO_2 layer, b) nucleation layer, c) epitaxial region, d) columnar growth.

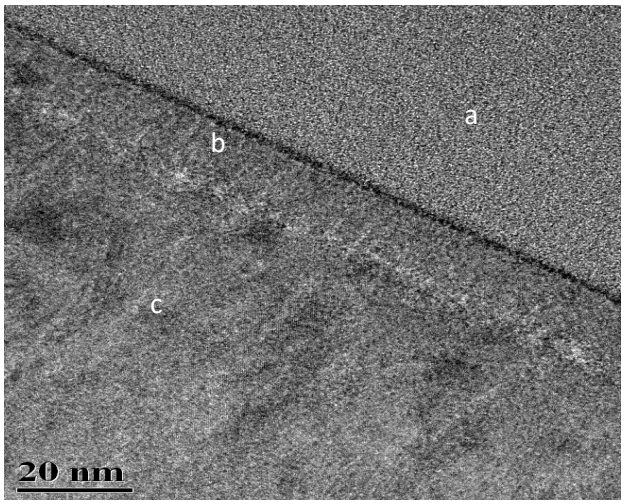


Fig. 4: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) SiO_2 layer, b) nucleation layer, c) epitaxial region.

X-TEM imaging were performed on Si/SiO_2 substrate having intrinsic thick polycrystalline and thin amorphous silicon film. From X-TEM imaging thickness of each layers are well observed. In figures 1&2, the SiO_2 layer 500nm (1a), the nucleation layer 20 nm (1b), epitaxial growth of 214 nm (1c) followed by columnar thick layer of 805 nm (1d), amorphous silicon layer 100 nm (1e), chrome layer 20nm (1f) and gold layer 80nm (1g) are shown.

Details view of top layers are shown in figure 2. A larger magnification image of each layer quantifies crystallinity. From figures 5 & 6 the expected polycrystalline arrangements are visible with a distinguished interfaces between oxides, nucleation and epitaxial layer is also observed. The top layers can be seen in figure 2(e) where amorphous region is visible.

Chromium film is visible in figure 2(f) where darkest portion is gold film 2(g). These films were deposited to avoid any damage on the silicon film during sample preparation process.

A diffraction pattern of polycrystalline silicon film is shown in fig 7. Diffraction pattern in fig 7c shows that the epitaxial layers are having both crystalline and polycrystalline phases whereas in figures 2d the polycrystalline patterns are visible. The three rings represents crystal orientation of (111), (220) and (311) planes those were publisher elsewhere [8,9]. From diffraction patterns of figures 7(c&d), it appears to be the interfaces between these layers which have mixed phase. This can be a contribution from polycrystalline layers.

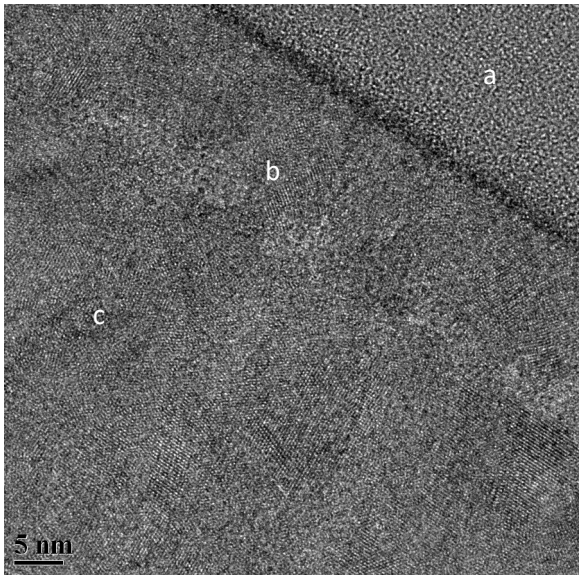


Fig. 5: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) SiO_2 layer, b) nucleation layer, c) epitaxial region.

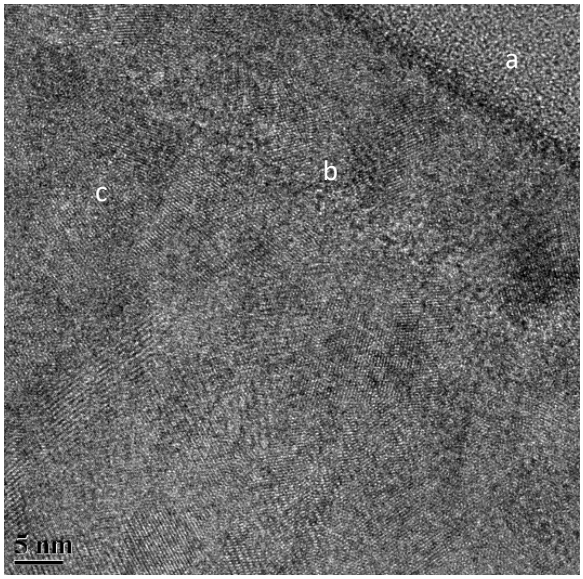
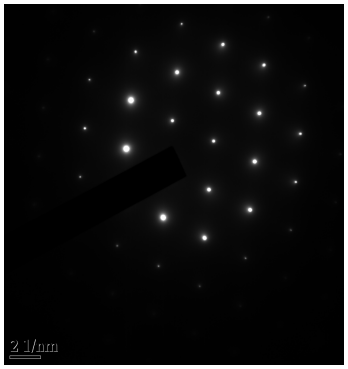
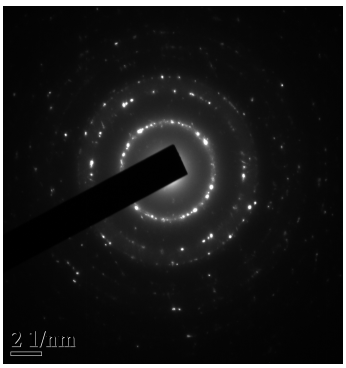


Fig. 6: Cross section TEM of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) SiO_2 layer, b) nucleation layer, c) epitaxial region.

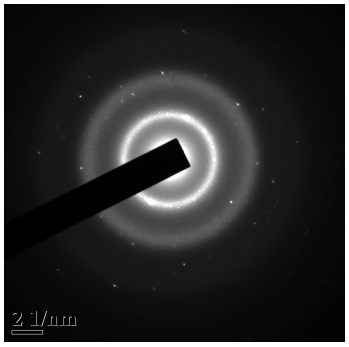


a) Silicon (100) wafer

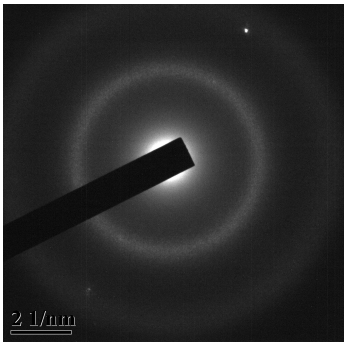
b) SiO_2 amorphous layer



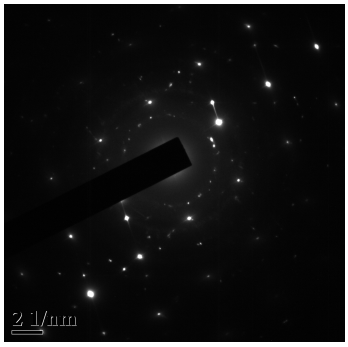
c) Epitaxial region



d) columnar growth



e) top amorphous layer



f) Top Gold-Chrome layer

Fig. 7: Diffraction pattern of HWCVD grown hydrogenated silicon on silicon (100) wafer. a) silicon wafer, b) SiO_2 amorphous layer, c) epitaxial region, d) columnar growth, e) amorphous silicon, f) gold chrome layer.

4 Conclusion

The observations using the cross sectional transmission electron microscopy of the structure of various layers of polycrystalline and amorphous silicon films intended for photovoltaic application has been presented. These observations allows us to evaluate amorphous and polycrystalline region as well as interfaces between layers from diffraction patterns. In the study we presented a technique for growing and defect annealing of intrinsic oriented crystalline poly Si film under atomic hydrogen soaking using HWCVD. Activation energy and photocurrent response analysis will help us to understand whether the films were suitable as absorber layer in photovoltaic devices or not. An initiative was taken to increase the thickness of films by increasing growth duration which will allow more light to be absorbed though the film got lifted due to its high stress and defects. An increase in hydrogen dilution may solve the issue. In that case process of increasing growth pressure in the chamber need to be studied.

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